LETTERS TO THE EDITOR

Measurement of nonuniform distribution of residual stresses in tempered glass discs

D. B. MARSHALL & B. R. LAWN

Department of Applied Physics, The University of New South Wales, Kensington

The strength of glass objects can be significantly increased by using physical or chemical tempering treatments to introduce residual compressive stresses into the surfaces.[1,2] Certain applications of tempered glass require the level of residual stress to vary across the surface: differentially tempered car windscreens[3] and cathode-ray tubes[4] are two important examples. Even where nonuniformity is not sought, it may be introduced inadvertently by the difficulty in maintaining constant treatment conditions over large surface areas. In either case it is an advantage for control purposes to be able to measure the stress distribution over any given tempered surface.

In this work we describe the application of a recently developed indentation technique[5] to the evaluation of nonuniform stresses in physically and chemically tempered flat discs, such as those used in safety goggles. Physical tempering was produced by conventional thermal quenching of heated discs in air jets and chemical tempering by an ion exchange process. An indication of the existence of surface variation in the residual stress could be obtained by viewing a given disc along its axis of symmetry between crossed polars. With physically tempered discs the familiar 'Maltese cross' was observed, as in Figure 1, whereas with chemically tempered discs no optical contrast was evident at all. The positive result with the thermally quenched discs appears to reflect an air-cooling configuration in which temperature gradients are established in the radius as well as in the thickness: a theoretical analysis by Kalman[6] has shown how the principal compressive stresses in an outwardly solidifying disc may be expected to fall off from the centre, with the decrease in the tangential component somewhat greater than that in the radial component. The negative result in the case of the chemically tempered discs appears to imply a process in which concentration gradients in the ion exchange species remain effectively negligible over the glass surface throughout treatment. However, while the optical method can be made quantitative, for example by measuring the birefringence averaged over the thickness of the glass at any point on the disc radius with a variable compensator, its information is limited to differences, and not absolute magnitudes, of the principal stresses. Thus, in the example of Figure 1, the measurement of a radially increasing birefringence, corresponding to a maximum difference between radial and tangential components of ≈ 18 MPa near the disc edge, tells us no more than that at least one of the principal stresses must be variable; the absence of any birefringence in the chemically tempered plates precludes any conclusion at all about variability, the radial and tangential components being everywhere equal.

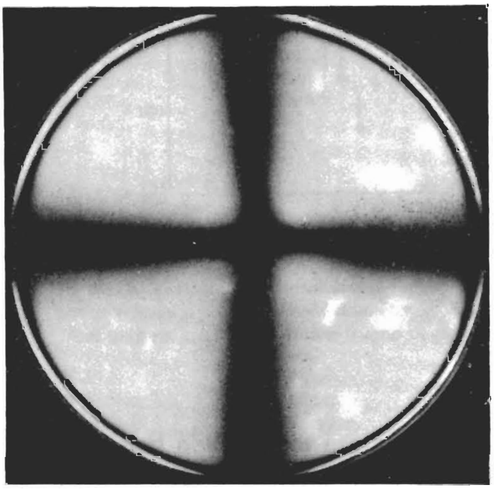

Figure 1. *Thermally tempered glass disc viewed between crossed polars, showing birefringence due to unequal radial and tangential components of residual stress*

The indentation technique for measuring residual stresses is not subject to such limitations. In its simplest form, the technique involves indenting tempered and untempered (control) surfaces with a standard Vickers diamond pyramid, at a prescribed load, such that a well developed pattern of 'median' half penny cracks is produced along the diagonals of the hardness impressions. Then from the comparative measurements one may evaluate the surface stress using the fracture mechanics relation[5]

$$\sigma = (K_c \pi^{\frac{1}{2}}/2c^{T\frac{1}{2}})[1 - (c^0/c^T)^{\frac{1}{2}}] \quad (1)$$

for cracks in an essentially uniform field, where c^T and c^0 are crack half lengths measured on tempered and untempered surfaces respectively, and $K_C = (0{\cdot}47 \pm 0{\cdot}07)$ MPa m$^{\frac{1}{2}}$ is the critical stress intensity factor (measured here in an independent set of calibration tests, based on an 'indentation/strength' procedure[7]). Point-by-point variations in surface stress may be mapped out most conveniently in terms of an appropriate array of indentations. Moreover, the Vickers impression can be produced in any specific orientation, so that the components of normal stress in any two mutually orthogonal directions on the surface may be measured simultaneously.

In using this technique, however, it needs to be emphasised that Equation (1) is not strictly valid when the stresses are not truly uniform over the entire area of the prospective cracks. Typically, the characteristic dimension of the median cracks in glass is $\approx 100\ \mu$m. Over such distances the variation in residual stress across the surface of a tempered plate is generally negligible (witness the data in Figure 2). On the other hand, the variation below the surface tends to be more severe, with fall off from maximum at the surface to zero over typical depths of ≈ 1 mm (i.e. $\approx$ one fifth of the plate thickness) for physically tempered glass and $\approx 100\ \mu$m (diffusion depth) for chemically tempered glass.[1,2] Consequently, stresses estimated from Equation (1) will tend to be low,[5] especially with the chemically tempered discs, although they remain a useful indicator of relative values for any given test surface.

In the present experiments indentations were made at intervals of 2 mm along a diameter of each disc, with the median cracks aligned parallel and perpendicular to the diameter to give the principal tangential and radial stresses respectively. The results are plotted in Figure 2. Whereas for the chemically treated discs the principal stresses show no systematic variation, for the physically treated discs both stress components diminish steadily to about one half their central value on approaching the edges. That the variation in the latter case is an effect intrinsic to the thermal tempering process rather than some spurious manifestation of, say, stress relaxation at the disc edge, is confirmed by the complete absence of any similar variation in the former case: any artifacts in the results would be expected to be common to all tempered discs. Again, with the thermally tempered discs there is an indication, notwithstanding the scatter in data, that the radial stress exceeds the tangential stress by about 10–20 MPa away from the disc centre, consistent with the earlier assessment from birefringence observations.

Such variations as shown in Figure 2 are of primary importance in the consideration of the strength of

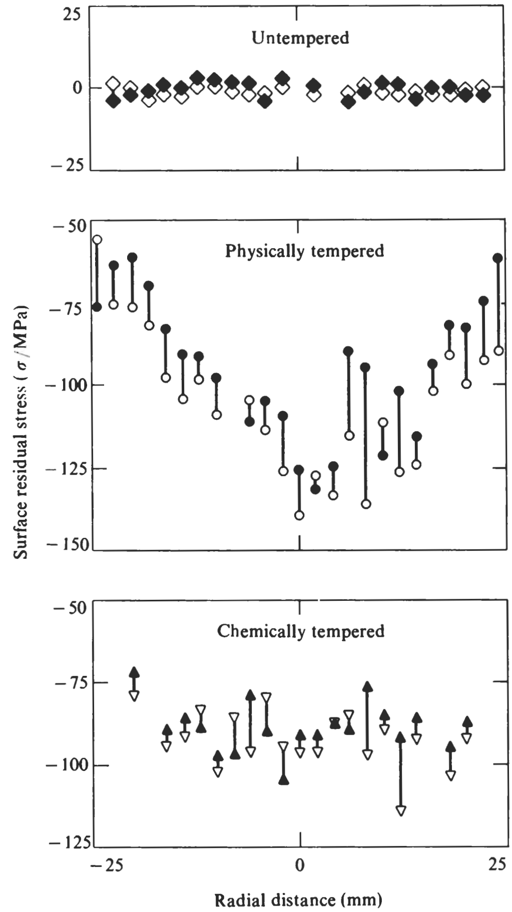

Figure 2. *Variation of radial stress (open symbols) and tangential stress (closed symbols) across diameter of glass discs as estimated from uniform stress indentation fracture equations*

tempered objects. For example, with thermally tempered safety lenses the beneficial effect of the residual compressive stresses will not be as great in a situation where the contact loading responsible for plate flexure is off centre, although this will be somewhat compensated by an attendant reduction in the intensity of flexural stresses generated.

Acknowledgements
The authors thank M. M. Lang for supplying treated glass discs. Funding was provided by the Australian Research Grants Committee.

References

1. Olcott, J. S. (1963). *Science* **140**, 1189.
2. Ernsberger, F. M. (1966). *Glass Ind.* **47**, 422.
3. Acloque, P. (1954). *Verres Réfract.* **8**, 243.
4. Gabor, D., Stuart, P. R. & Kalman, P. G. (1958). *Proc. Instn elect. Engrs* **105B**, 581.
5. Marshall, D. B. & Lawn, B. R. (1977). *J. Am. Ceram. Soc.* In press.
6. Kalman, P. G. (1960). *J. Am. Ceram. Soc.* **43**, 313.
7. Marshall, D. B. & Lawn, B. R. (1977). *J. Am. Ceram. Soc.* In press.